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A precision curved crystal X-ray and gamma-ray spectrometer

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With 9 figures in the text

1. Principles of the spectrometer construction

The spectrometer to be described on the following pages was designed for precision measurements of wavelengths in the X-ray spectra of heavy elements. It is essential to have an instrument with high luminosity and high dispersion for this purpose. These requirements are met by the curved crystal spectrometer, and, for the relatively short wavelengths in question (100–700 XU), the transmission type is the only one available.

Two different arrangements with the same fundamental geometry are then possible, the Cauchois and the Du Mond arrangement, shown in Figs. 1 and 2. In both cases the focussing condition implies that the slit source of radiation and the "image" formed by diffraction in the internal atomic planes of the crystal, should both lie on the Rowland circle according to the figures. The diameter of the Rowland circle equals the radius of curvature of the bent crystal. The angle (θ) determining the wavelength (λ) of the diffracted radiation is the Bragg angle given by the well-known relation:

$$\lambda = 2d \sin \theta. \tag{1}$$

d stands for the lattice spacing of the atomic planes in the unstressed zone of the crystal. The fact that the crystal fails to coincide with the Rowland circle at its extremities gives rise to a small aberration which can be corrected for.

The geometrical difference between the two types lies in the fact that in the Cauchois type the slit source is virtual and the image real, while the opposite is the case in the Du Mond type. This means that photographic registration is possible only with the Cauchois type. But as the photographic method is restricted to relative measurements, it was abandoned for the present purpose in favour of the point by point method of exploring the spectra, with the use of an electronic detector.

Comparing the two arrangements from the point of view of the intensity of the spectral lines, the Du Mond type is favoured when the source of radiation is small in size. Each point on the extended source in the Cauchois arrangement emits radiation which has a given wavelength and which is diffracted by the crystal, only in a small solid angle. This solid angle is limited by the angular region around the Bragg angle where diffraction of the actual wavelength takes place owing to the finite

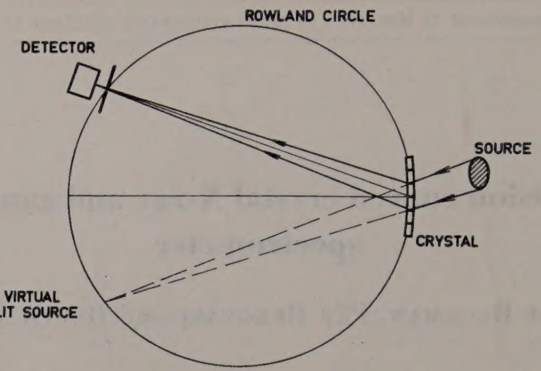

Fig. 1. The Cauchois arrangement of a curved crystal transmission spectrometer.

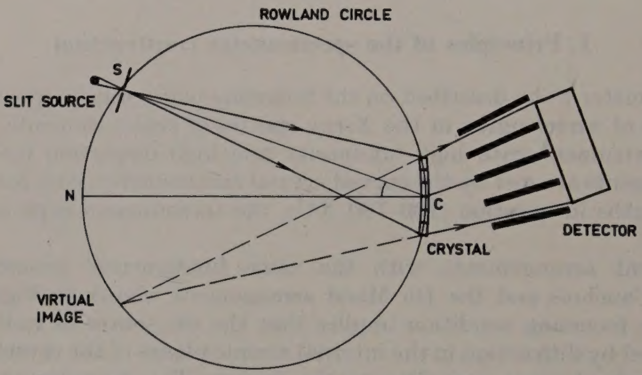

Fig. 2. The Du Mond arrangement of a curved crystal transmission spectrometer.

extent of the diffraction pattern. In the Du Mond arrangement each point on the source emits effectively in the whole solid angle given by the aperture of the crystal. Here, however, the extent of the source must be defined by a slit in order not to contribute unduly to the width of the measured spectral lines. It is also apparent that this arrangement requires a very accurately curved crystal if the gain in intensity is not to be accompanied by a loss in resolution due to focal aberration.

In one more respect the Du Mond arrangement is advantageous. It is possible to shield the detector from the undiffracted, direct beam of radiation with a baffle or a "collimator". This implies that higher energies can be attained with a much better signal to background ratio than with the Cauchois arrangement.

For the construction of a Du Mond spectrometer, we must consider which parts of the instrument must be moved with high precision and which parts require less exact motion as the Bragg angle is changed. Of the three components: detector, crystal, and source, at least two must be given an angular motion around an axis thorugh C. The detector, which has to collect the diffracted radiation requires no precise motion, at least if the angular resolution of the baffle is low. A low resolution can be allowed in this case as only the relatively low energies below 350 keV are to be measured. The angle SCN in Fig. 2 is the all-important Bragg angle which must be measured with high precision. It is therefore essential that of the two chords SC and NC of the Rowland circle, defining this angle, one should be fixed and only one moving as the angle is changed. It is our opinion that mechanical links and other devices intended to secure a motion of the two chords while at the same time an exact indication of the angle between them is furnished, are very difficult to realize [2, 5, 13]. The source cannot be given an angular motion in our case, as it is the target of an electrostatic generator. This is also the case for gamma spectrometers using the radiation from in-pile sources [12]. But also as far as gamma spectrometers with conventional sources are concerned, the main bulk of the lead shielding is preferably concentrated around the source, making it less mobile than the detector assembly. It is also preferable, in principle, to work with a fixed source slit. The Bragg angle is then determined by the angle through which the crystal normal CN has been turned, and this angle can be measured with high precision with an angular scale. The detector should be moved double the angle which the crystal is rotated and around the same axis in order to keep in the correct position relative to the source slit and the crystal. A wavelength measurement will consist of the delineation of a spectral line in the two mirror-symmetric positions of the spectrometer. The angle which the crystal is turned from one position to the other is the double Bragg angle.

There is one point which requires our attention in this arrangement. In order to remain on the Rowland circle as the crystal is rotated, the source slit must slide in the direction of the central ray (a line through the geometrical centers of the slit and the crystal). This motion is, however, easy to accomplish as it is not an angular but a linear motion and of comparatively small amplitude. In our case it is not necessary to move the source of radiation itself, but only the limiting slit. The slit may be kept in the same position while the spectrometer is working in the narrow wavelength regions comprising a spectral line or even a group of lines. During the delineation of a spectral line in the two spectrometer positions, the slit is of course kept fixed. The departure of the slit from the Rowland circle is expressed by:

$$R(1-\cos\theta) \tag{2}$$

(R being the diameter of the Rowland circle) and therefore varies very slowly for Bragg angles smaller than 10 degrees. The correct position of the slit for each case is easily and accurately found by the Hartmann tests described below.

For the wavelength determinations the question now arises which value to adopt for the spacing (d) of the atomic planes, in equation (1). For quartz, which is the material used here, accurate measurements have previously been made of the spacings of the different atomic planes with the use of certain well-established X-ray lines. The appropriate procedure for us, however, must be an independent measurement of our spacing constant as it is used in the spectrometer, employing these X-ray lines. There is a great advantage in this, pointed out by Du Mond [5]. All systematic errors in the wavelength measurement which are linearly dependent on the wavelength, are automatically cancelled, the correction being included in the measured value of the spacing constant, which thus becomes a constant characteristic of the instrument rather than of the diffracting crystal. X-ray lines with well-established wavelength values, suitable for this measurement, are given by Sandström [14]. They include the $K\alpha_1$ lines of molybdenum, silver, antimony, and tungsten.

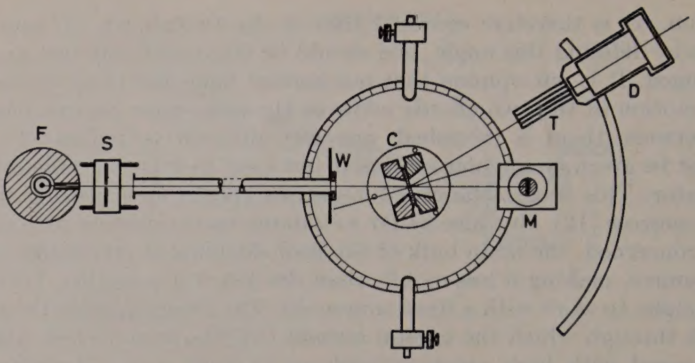

Fig. 3. Outline drawing of the spectrometer. For explanation see the text.

2. Description of the spectrometer design

2.1. General

An outline drawing of the spectrometer is given in Fig. 3, and a view of the instrument, except of the slit, is found in Fig. 4. The X-ray beam travels from the left to the right in both pictures. From the source F, limited by the slit S, the X-rays

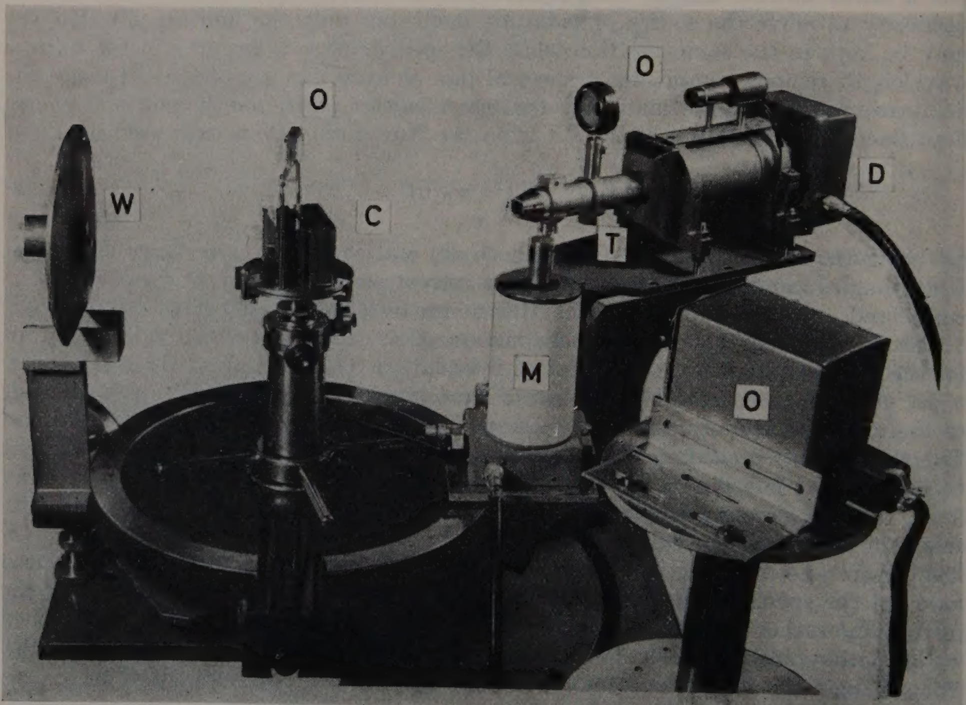

Fig. 4. View of the crystal holder, circular scale and detector arrangement.

pass through a 1.8 meter long brass tube, encased in 5 mm lead. They emerge from a brass aperture *W* and strike the crystal *C*, which is clamped in steel blocks, standing on the spectrometer table. The main part of the beam goes undiffracted through the crystal and hits the monitor scintillator *M*. The diffracted radiation is collected through the collimator tube *T* by the detector *D*, a scintillation counter.

The spectrometer table with the crystal can be rotated and the angle measured with reading microscopes on a precision scale. This scale, together with its associated components has been taken over from the tube-spectrometer described by Siegbahn–Larsson [10]. Only three components in the original design have been rebuilt: the spectrometer table, the lever and the screw for turning the scale in small steps, and the mechanism locking this lever to the conical support of the scale. The detector *D* rests on a carriage by which it can be turned around the same axis as the crystal and the scale. The optimal position of the detector relative to the crystal and the slit is ensured with the aid of an optical system *O*.

The low energy limit of the spectrometer is set by the increasing absorption in the crystal. It is about 700 XU (17.5 keV) and corresponds to the *K* spectrum of molybdenum. For X-rays, the upper limit is determined only by the position of the monitor scintillator, which must be placed sufficiently far from the crystal, so as not to intercept the diffracted radiation. This limit is above the *K* spectrum of plutonium. For gamma sources, where no monitor is required, the upper energy limit is set by the reflection power of the crystal which decreases as the square of the wavelength. At present, however, the limit is determined by the simple construction of the baffle, and is about 350 keV.

Detailed descriptions of the more important parts of the instrument are given in the following sections.

2.2. The source and the slit

As X-ray tube the acceleration column of a van de Graaff generator is used. This has previously been described by Beckman [2]. A few improvements have been made, however, the most important one being the stabilization of spray- and emission current. Thus a more stable X-ray output is achieved. The generator has been running at about 550 kV and 40 μA.

The target arrangement is the same as that described by Beckman [2]. The electron beam passes two crossed magnetic fields and a small circular aperture, connected to ground, before striking the electrically insulated target plate. Any deflection of the electron beam is registered as a decrease in target current, and this is compensated for with the magnetic fields. Thus the position of the focal spot is kept fixed. The target materials W, Ag, and Mo were available as 1.5 mm thick circular plates, 20 mm in diameter, whereas Sb was soldered on to a copper plate of the same dimensions.

Because of the short wavelengths, the slit defining the source must be made of a heavy element. The jaws of the slit consist of two gold plates, 1 mm thick, soldered to two pieces of silver, also 1 mm thick. The slit is mounted in a ring, to which a short lever is attached as seen in Fig. 5. By means of two screws (*A*), the slit can thus be adjusted parallel to the diffracting atomic planes of the curved crystal. The slit is further mounted on a slide providing the required motion along the direction of the central ray. The slide which is provided with an index, moves on two steel bars, resting on a graduated brass plate. This plate can be rotated around a vertical

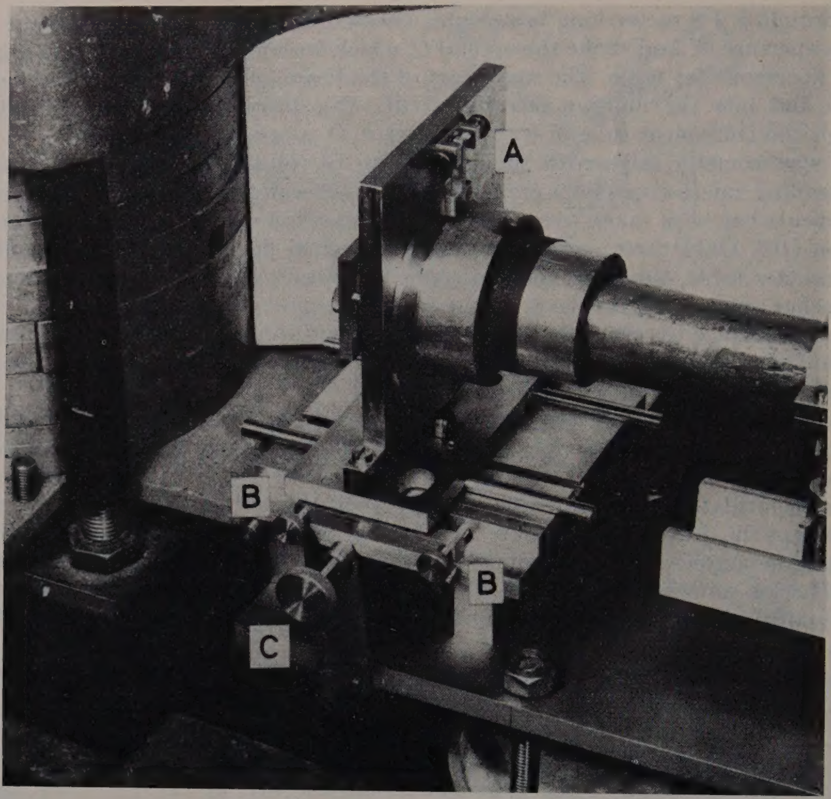

Fig. 5. View of the slit arrangement.

axis with the screws (B), allowing a correct alignment of the steel bars. Furthermore, it can be moved in a direction perpendicular to the spectrometer axis by means of two screws, one of which is visible in the figure (C).

2.3. The curved crystal

The crystal is made of quartz, and as diffracting atomic planes the (310) planes have been chosen because of their high reflecting power and suitable lattice spacing. The plate is cut so that its largest faces are parallel to the trigonal axis and perpendicular to the (310) planes. The edges of the plate are then cut with the base edge perpendicular to these planes. The crystal plates used here are prepared to the above specification by Steeg & Reuter, Bad Homburg.

A radius of 2 meters was chosen for the curvature of the crystal. There are two methods of bending the crystal lamina into the desired cylindrical shape, the "method of freedom" and the "method of imprisonment". The first method, described by Brogren [3], employs four parallel steel bars, between which the crystal lamina is freely sprung. A plate-holder of this type has the advantage of being easy to manufacture. In the Cauchois type spectrometer it has been extensively used. There, however, only a small part of the crystal is used, corresponding to the area of the source of radiation. In the Du Mond type the whole surface of the curved crystal is used and higher demands must be put on the quality of the crystal profile. For this reason the method of imprisonment has been perfected by Du Mond. The crystal is forced into cylindrical shape between two rigid surfaces, one concave and the other convex, accurately ground to cylinders of the desired radius on hardened steel blocks.

For testing the quality of a curved crystal and determining its focal point, Hartmann tests [5] are indispensable. In the present case, they are made with the crystal mounted in the spectrometer, and consist of a delineation of a spectral line with the use of different parts of the crystal. The source slit is then moved until the point of closest overlapping of the profiles is found. This optimal position of the source slit can be established with an accuracy of a millimeter. The maximum spread in line position at this point is a measure of the focal aberration of the crystal.

During the preliminary stages of the spectrometer construction, the method of freedom was attempted. The size of the quartz plate was $25 \times 15 \times 1$ mm^3. It was bent under optical control, and care was taken to ensure a symmetrical profile. The aperture in the crystal holder was 10×14 mm^2. When the source slit was placed at the point of best focus, a half-width of the W $K\alpha_1$ line of 35″ was obtained, corrected for slit width: 33″. When only a part of the total aperture, 5×10 mm^2 in size, was used, this figure was reduced to 27″. It was confirmed by the Hartmann tests that the crystal had certainly acquired a symmetrical profile but showed a pronounced saddle-shape. The unsatisfactory result was evidently due to the method employed, which hence was abandoned.

For a successful application of the method of imprisonment it is essential that the clamping blocks be ground and lapped into cylindrical surfaces of high precision. This was done by AB Svenska Kullagerfabriken (SKF) in Gothenburg, to meet our rigorous requirements. The material used was steel, hardened before the grinding. The generatrices of the cylindrical surfaces were ground accurately at right angles to the top and bottom surfaces of the blocks. The aperture for admitting the X-radiation is tapered, with no supporting ribs and a minimum area of 15×20 mm^2. The dimensions of the crystal lamina are $1.5 \times 35 \times 45$ mm^3.

The bending procedure will be briefly described. The convex clamping block was screwed on to the spectrometer table. Next to it, tangential to the cylinder surface, a steel parallel was placed. The crystal plate was then balanced with its base edge on the steel parallel and one of its large faces against the convex surface. All surfaces were kept dust-free and a thin oil film was applied to reduce friction. The concave clamping block was provided with a 0.5 mm thick rubber gasket on the curved surface, and was then pressed against the crystal and the convex block. The pressure was applied by four screws, threaded in the convex block and passing through the concave block. The screw heads were not in direct contact with the concave block. Instead, the pressure was exerted via spiral steel springs. This method is the same as that used by Du Mond. The bending procedure was controlled optically, and particular attention was paid to effecting an even pressure from all four screws.

For the Hartmann tests the crystal aperture was divided into three parts. The three corresponding line profiles of W $K\alpha_1$ were brought to the point of closest ovelapping by varying the source slit position. Two of the profiles coincided, and the third was out of position by 3″. This is equivalent to a focal aberration of 0.03 mm, which is a satisfactory result. The half-width of the W $K\alpha_1$ line with the whole crystal aperture employed, was 23″. If this is corrected for slit width, 20″ remain. This figure embodies the spectrometer resolution and the natural line width. The latter is known to be about 12″ in this scale. The minimum resolution for the instrument (with an infinitely narrow slit) at this energy is accordingly about 0.05 %. The dispersion is determined by the spacing of the (310) planes and is 0.0114 XU/sec. of arc for small Bragg angles.

2.4. The angular scale

The angular scale is identical with the scale used in the Siegbahn–Larsson tube-spectrometer [10]. It is known to be of very high quality, and was last used for precision measurements in 1953.

The eccentricity of the scale was first measured. The scale ring was then adjusted relative to its conical shaft with four centering screws and the eccentricity was reduced until it was beyond detection.

For the present measurements a 35°-interval of the scale would be sufficient. To select this interval and to make a general survey of the scale, we proceeded in the following way. The range of sight in a reading microscope comprises 1° of the scale. With the eyepiece micrometer the length of a 0.5°-interval was measured in terms of the micrometer screw. This was repeated for all 0.5°-intervals around the scale. The accumulated sum of these intervals was plotted in a diagram as a function of the scale position. A statistical distribution of the scale divisions around their correct positions will show up as a scattering of the points around a straight line. A systematic deviation in homogeneity or a discontinuity in the scale will show up in the diagram as a break in the straight line or a change of its slope, gradually or abruptly.

A 35°-section of the scale was found, satisfying the following demands:

1. The scale showed no signs of corrosion and the divisions were clearly defined and easy to read.
2. The division of the scale was of high homogeneity; the lengths of the 0.5°-intervals showed a mean square deviation of 0.2″ from their mean. The points in the diagram described scattered about a straight line.
3. The mean length of the intervals agreed closely with the mean obtained for the whole scale.

As a systematic discontinuity might be concealed in the statistical deviation, although this seemed improbable, a further test of the scale section was performed. The W $K\alpha_1$ line, which corresponds to a double Bragg angle of about 10°, was measured with the use of three different, slightly overlapping parts of the 35°-section. The measured Bragg angles are given on page 434 and show good agreement.

Finally, the close correspondence of the four calibration measurements with the use of different reference X-ray lines, gives strong evidence in favour of the high quality of the scale.

2.5. The detector and the monitor

The detector is a NaI(Tl) crystal scintillation counter, combined with a single channel discriminator. This makes it possible to maintain a very low background, as the radiation of all energies except that of the X- or gamma-ray line measured is discarded. The pulses are recorded by a decade scaler. The counting rate never exceeded 3000 counts per second. Thus no problem of dead-time corrections entered as the pulses from a scintillation counter are very short (≈ 1 μsec.).

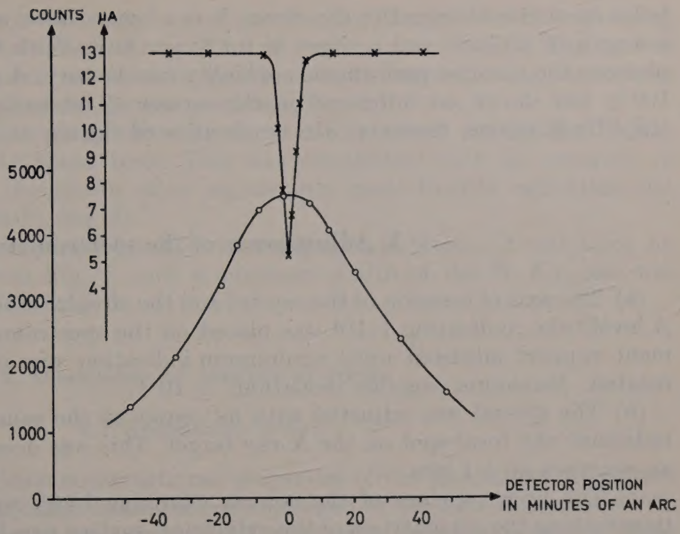

Fig. 6. Transmission curve of a collimator using 3 lead partitions (○), together with the signal given by the optical control system (×).

The scintillation crystal, photomultiplier and pre-amplifier are surrounded with lead shielding and the whole system is movable around the axis of rotation of the spectrometer. A tubular collimator, made of copper encased in lead, having a rectangular aperture in the entrance end, is used. The aperture has the same area as the opening in the crystal clamping blocks. Such a simple collimator is sufficient for measurements of Bragg angles larger than half the angular aperture of the crystal viewed from the slit. A more elaborate collimator, in which thin lead walls are introduced, preventing the direct beam from striking the detector crystal, had to be used in the present spectrometer for energies of the examined radiation greater than 200 keV. This more complicated collimator, which was used when the 328 keV gamma-line of Ir^{194} was measured, consists of three vertical partitions of uniform wall thickness (1 mm) with the openings between the partitions tapered.

The geometry of the spectrometer requires that an angular displacement $\Delta\theta$ of the crystal is followed by a rotation $2\Delta\theta$ of the detector. This will be accounted for by means of the optical arrangement seen in Fig. 4. A slit-lamp and a collecting lens are placed on the collimator. The convergent beam of light is reflected in a plane mirror, fixed relative to the spectrometer table, and is focussed on a stationary narrow slit in front of a photo-cell. Fig. 6 shows the intensity of the X-rays as well as the amplified current from the photo-cell for different positions of the detector. Apparently the detector adjustment is critical only within ± 2 minutes of arc. With the optical system, the detector position can be ascertained with an accuracy of ± 40 seconds. A spectral line never extends over more than 80 seconds of detector motion. Therefore, the detector is kept fixed during each delineation.

The monitor is a scintillation counter. The crystal is a flat NaI(Tl) plate, facing the direct beam. It has the same area as the cross-section of the beam, and a thickness of 3 mm. It is mounted in a lucite light guide, surrounded by 3 mm of lead. The counter employs a separate linear amplifier and single channel discriminator, and the pulses are recorded by a predetermined count scaler. This scaler also gives pulses to switch on and off the electronics registering the X-ray intensity. The monitor pulse spectrum obtained in the direct X-ray beam shows a plateau at 165 keV with a length of 50 keV and a slope of 0.1 % per keV. With the channel fixed on this plateau, the monitor performance is highly satisfactory. A change in counting rate of 100 % has shown an influence on the measured intensity of less than 6 %. Such large fluctuations, however, are never allowed during the measurements.

3. Adjustments of the spectrometer

(a) The axis of rotation of the crystal and the circular scale was adjusted vertically. A level tube, indicating $1:10^4$ was placed on the spectrometer table and the instrument support adjusted until a minimum indication was obtained as the scale was rotated. Maximum angular deviation: $\pm 10^{-4}$.

(b) The crystal was adjusted with its center at the same height as the source of radiation; the focal spot on the X-ray target. This was done with a water level with an accuracy of ± 1 mm.

(c) The upper surface of the convex clamping block was adjusted horizontally, thus making the generatrices of the cylindrical surface parallel to the axis of rotation. This was done with the three screws of the spectrometer table with use of the level tube. Maximum angular deviation: $\pm 10^{-4}$.

(d) The crystal was centered on its axis of rotation. Two motions of the spectrometer table at right angles to each other and to the axis are provided with two centering screws. A microscope with an eyepiece scale was placed above the crystal, and the upper edge of the crystal lamina was observed. By successive adjustments of the microscope and the centering screws as the scale was turned 180°, centering was obtained in the two directions of the cross-table. In the radial direction of the curved crystal the centering was made to within 0.03 mm. To this must be added the influence of an error in the adjustment of section (*c*), as one is only looking at the top of the crystal lamina, which might be inclined relative to the axis of rotation. This makes the maximum uncertainty in the radial direction equal to ± 0.04 mm. In the direction tangential to the curved crystal a centering within ± 0.5 mm was obtained.

(e) The monitor scintillator was centered in line with the crystal and the source of radiation.

(f) The source slit was adjusted in line with the geometrical centers of the crystal and the source of radiation. A wide lead slit was placed in front of the crystal and the counting rate of the monitor was recorded for different positions of the slit. This gave information about the X-ray illumination of the crystal. The position of the source slit was then adjusted with the screws (C) in Fig. 5 until a uniform illumination was obtained.

(g) The motion of the source slit parallel to the direction of the central ray was adjusted. The slit was illuminated with a lamp and the optical image formed by the curved crystal lamina was observed with a microscope as the slit was moved. With the screws (B) in Fig. 5 it was obtained that the image displayed no appreciable transverse motion. Thus it is ensured that the X-ray illumination of the crystal is uniform for the different slit positions which are to be used.

(h) The detector and the baffle were adjusted to the right height. Then the correct position of the detector relative to the crystal and the slit was sought. This was done with X-rays, the crystal being in the position for Bragg reflection of a $K\alpha_1$ line. The detector was moved and the position of peak intensity found. The optical system, ensuring that the correct detector position can be maintained as the crystal is rotated, was then adjusted to fit this position. (Fig. 6.)

(i) The optimum position along the central ray of the source slit for the W $K\alpha_1$ line was obtained by Hartmann tests. This was established with an accuracy of ± 1 mm. The position of the slit for other wavelengths could then be calculated and was checked experimentally (eq. 2).

(j) The slit was adjusted parallel to the reflecting atomic planes. It was tilted by means of the screws (A) in Fig. 5 until a minimum width of the W $K\alpha_1$ line was obtained.

4. Discussion of sources of errors

4.1 Systematic errors

These are due to intrinsic constructional properties of the instrument, to imperfections of the angular scale and reading microscopes and to the limited accuracy with which the spectrometer has been adjusted. All systematic aberrations with a linear dependence on the wavelength are automatically corrected for, since they are included in the instrumental constant. (Cf. page 421.)

a. *Aberration due to the inexact focal properties of the spectrometer type*

As the curved crystal does not coincide with the Rowland circle, an aberration, which has been discussed by Du Mond [5], is introduced. It gives a line broadening and a line shift $\Delta\lambda$ towards longer wavelengths, which, for an aperture angle $2\alpha \ll 1$, is given by the formula:

$$\Delta\lambda \approx \frac{\alpha^2}{4}\lambda.$$

The shift is seen to be linear with wavelength. It has a maximal value of 0.005 XU at 700 XU.

b. *Vertical divergence*

We assume that the central ray of the X-ray beam, AO in Fig. 7, going at right angles with the axis of rotation of the crystal, is making a glancing angle θ with the diffracting atomic planes. The angle of incidence relative to the normal NO is then $(90° - \theta)$. The ray BO of vertical divergence φ is a ray lying in the vertical plane which contains the central ray but making an angle φ with it. BO makes an angle of incidence of $(90° - \theta')$. The above three angles enter as arcs into a right spherical triangle which is solved with Neper's rule:

$$\cos(90 - \theta') = \cos(90 - \theta)\cos\varphi.$$

The angle φ is small. Introducing $\Delta\theta = \theta - \theta'$, we obtain:

$$\Delta\theta \approx \frac{\varphi^2}{2}\tan\theta. \qquad (3)$$

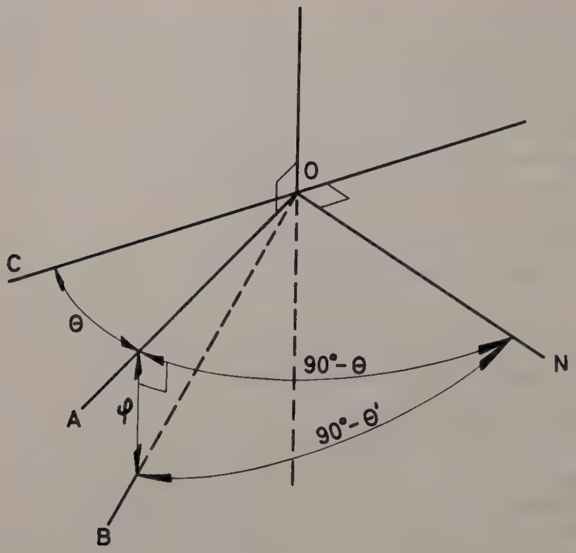

Fig. 7. Geometry determining the effect on the Bragg angle of certain instrumental aberrations.

The expected wavelength shift of a spectral line, $\Delta\lambda$, is about half as much as the shift corresponding to the maximum vertical divergence angle φ_m. Changing from angle to wavelength, using the Bragg equation (1), we obtain:

$$\Delta\lambda \approx \frac{\varphi_m^2}{4}\lambda.$$

The shift is linear with wavelength. When the angle φ_m is computed, we take into account that the focal spot is adjusted to the same height as the centre of the crystal within certain limits of accuracy. An error in this adjustment has the same effect as an increased value of φ_m.

The maximum shift according to the above is 0.005 XU at 700 XU.

c. *The angular scale*

The eccentricity of the angular scale has been reduced to a negligible amount. It has also been verified that the section of the scale used exhibits no systematic discontinuities. The scale divisions, however, show deviations from their correct positions, which show a statistical distribution, the mean square deviation of which is 0.2″. When the angular position of a spectral line is determined, readings on 5 consecutive scale divisions are taken. The statistical accuracy of the mean Bragg angle with respect to the above deviations is thus less than 0.1″.

d. *The reading microscope*

The screw moving the parallel hairs of the eyepiece scale in the microscope is of high quality. It moves under the influence of no external forces other than the light torque required to move the light carriage with the hairs. The drum at the head of the screw is divided into 120 divisions. 1 division equals 0.99922 seconds of arc. To prevent the influence of backlash the screw is always moved in one direction when used. The effect of screw errors is diminished as readings are taken on 5 consecutive scale divisions on the angular scale. In particular, periodic errors reduce in importance as the screw is turned $2\frac{1}{2}$ turns between the divisions, these being $5'$ apart. In this way the error introduced by the screw is kept lower than $0.1''$.

e. *Orientation of the diffracting atomic planes of the crystal*

The axis of rotation has been adjusted vertically with very high accuracy, and it can also be assumed to be at right angles with the central ray as the error in this adjustment has been included in the vertical divergence effect. The atomic diffracting planes should be parallel with the axis of rotation, which is vertical, and intersect diametrically on the Rowland circle, in point N, Fig. 2. The latter condition has been fulfilled with sufficient accuracy as the Hartmann tests have determined the focal aberration as negligible.

If the atomic planes make an angle ψ with the vertical line, an error is introduced in the Bragg angle. Referring to Fig. 7 again, NO is this time the central ray, and BO the normal of the inclining atomic planes. From the zero position, with the atomic planes parallel to NO, the crystal has been turned anticlockwise the angle θ, and this is the angle measured on the angular scale. The angle of incidence, however, is $(90° - \theta')$, the complement of the Bragg angle. Thus we obtain the same geometry as for vertical divergence. A spectral line is shifted by the amount $\Delta\lambda$ corresponding to the deviation in Bragg angle $\Delta\theta$, given by equation (3).

$$\Delta\lambda \approx \frac{\psi^2}{4}\lambda.$$

The shift is linear with wavelength. To obtain its magnitude we consider first the orientation of the atomic planes relative to the edges of the crystal lamina, where an error of at most 5 minutes of arc is given. The alignment of the crystal lamina with the convex clamping block is accomplished with a steel parallel. If we allow for an error of 10^{-3} in the alignment of the crystal plate relative to the axis of rotation, we obtain a maximum value for $\Delta\lambda$ of 0.003 XU at 700 XU.

In the above discussion we have assumed that the diffracting atomic planes were parallel to the generatrices of the cylindrical surface of the convex clamping block. An error in this respect will primarily give unsatisfactory focal properties of the crystal. The aberration introduced in the wavelength measurements is, however, difficult to analyze. But as the Hartmann tests and the widths of the spectral lines show an excellent focussing of the crystal, the wavelength errors must be negligible.

f. *Orientation of the axis of rotation of the scale and the crystal*

The axis is vertical within limits of negligible influence. It should also pass through the unstressed zone, that is the middle of the crystal lamina in the direction of its thickness. An error (a) in this adjustment has a direct influence on the Bragg angle (θ). Simple geometrical considerations yield the formulae:

$$\Delta\theta \approx \frac{a}{R}\operatorname{tg}\theta; \qquad \Delta\lambda \approx \frac{a}{R}\lambda.$$

Here R is the radius of curvature of the crystal. The shift is linear with wavelength. Its maximum value is 0.01 XU at 700 XU.

It is desirable that the axis of rotation should pass through the geometrical center of the crystal also in the direction tangential to the curved surface. An error in this alignment has no influence except as a small contribution to the aberration of section 4.1.a.

g. *Position of the source slit*

If the crystal is uniformly illuminated an error in the slit position implies a broadening but no shift of the spectral lines. As the slit is moved along the central ray for different wavelength regions, the uniform illumination has been ensured. A spectral line is always delineated in the two symmetric positions of the instrument, and during this procedure, the slit is of course kept fixed. Thus the slit position bears no significance on the determination of the Bragg angles.

h. *Temperature correction*

The spectrometer room is "air-conditioned" (for the purpose of ensuring stability of the van de Graaff generator) for constant humidity and temperature. The temperature was measured in the air close to the curved crystal, and exhibited only small variations, always less than $\pm 0.3°$ during a line measurement.

The thermal expansion coefficient of quartz in the given direction has been taken as 14.5×10^{-6} [9]. All Bragg angles have been reduced to 18.0°C.

4.2. Accidental errors

a. *Accuracy of the angle measurement*

An individual reading of the scale position is made with a maximal error of $0.5''$ and a mean square deviation of $0.2''$. At least 10 to 15 such readings determine the position of a spectral line, which thus is affected less than $0.1''$.

b. *Line position*

There are different conventions regarding the definition of "the wavelength" of a spectral line. The point of maximum intensity, the mean diameter of horizontal chords, and the centroid have been suggested. All our spectral lines have shown a symmetrical shape within the experimental errors, and the above definitions then coincide. As the most practical method we have chosen to establish the diameter of horizontal chords. Due to statistical fluctuations in the intensity and the angle measurements and different possiblilities of fitting a curve to the experimental points, we estimate a maximum error in the position of the diameter, Δ, as 1/40 of the width of the line at half maximum intensity. This is in good agreement with the observed reproducibility.

4.3. Assigned errors

If n pairs of a spectral line are measured, the mean Bragg angle is determined by $2n$ independent measurements, each of which carries the accidental error Δ. The accuracy of the mean is then given by the expression:

$$\frac{\Delta}{\sqrt{2n}}.$$

To the figure thus obtained we have added 0.15″, estimated to account for non-linear systematic errors. The resulting limits of accuracy are given below for each line. For the case of W $K\alpha_1$, which was measured with three different parts of the scale, the systematic error has been estimated to be less than 0.05″.

5. Measurements

5.1. Determination of the spectrometer constant d

Sandström (14) has recommended the $K\alpha_1$ lines of tungsten, antimony, silver, and molybdenum to be used as reference lines. A determination of the Bragg angles for these lines was made with the spectrometer. A typical recording of a line pair is shown in Fig. 8. The results are tabulated below. The corresponding values of the constant d in eq. (1) were calculated, with the use of the wavelength values given by Sandström. The values of d are found in Table 5.

Results: See Tables 1–5. θ_t = measured Bragg angle at temperature t; θ_0 = corrected Bragg angle at 18°C.

As seen, the value obtained for silver shows a slight discrepancy. The given wavelength value was therefore examined more closely. A discussion of the determinations of this line up to 1936 was given by Ingelstam [8], who used this line as a reference. He found that a simple mean of the five different measurements made, 558.241 XU, was most compatible with the other reference lines he used. Shortly afterwards, in 1937, a measurement by Elg [6] was made, yielding the slightly lower value 558.235 XU. In 1951 a renewed determination was made by N. Nilsson with the tube spectrometer [11]. She found the value 558.252 XU. This is in good agreement with the

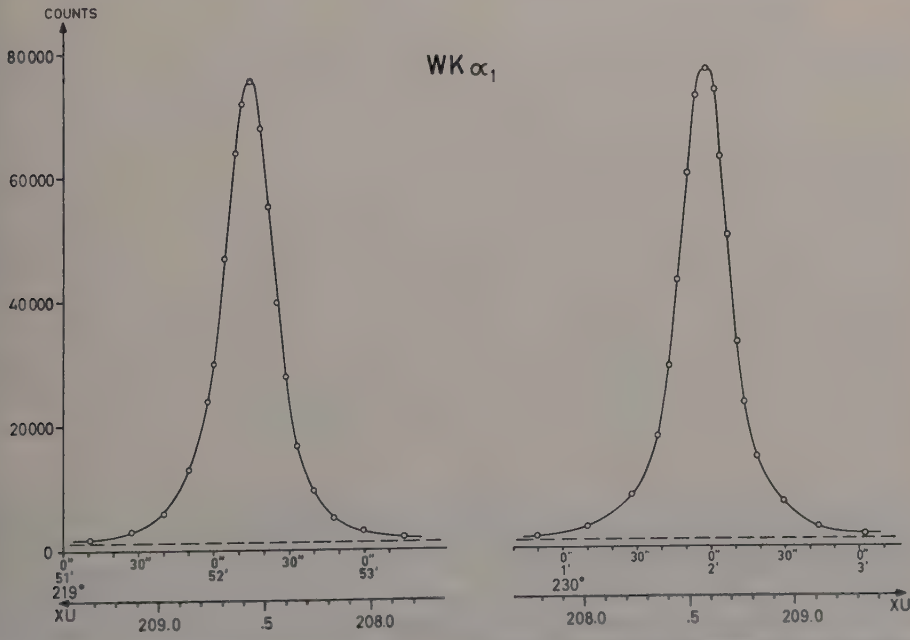

Fig. 8. W $K\alpha_1$ delineated in the two symmetric positions of the spectrometer. Line width 23″.

Table 1. W $K\alpha_1$.

The three sections of the scale used are labelled I, II and III.

Scale section	θ_t	t °C	θ_0
I	5° 4′ 50.3″	22.1	5° 4′ 51.4″
	50.5	21.5	51.4
	50.4	21.3	51.3
	50.6	21.1	51.4
II	5° 4′ 50.6″	20.9	5° 4′ 51.4″
	50.4	21.3	51.3
III	5° 4′ 50.5	21.4	5° 4′ 51.4″
	50.9	21.3	51.8
		Mean:	5° 4′ 51.4″
		Error:	±0.2″

Table 2. Sb $K\alpha_1$.

θ_t	t °C	θ_0
11° 29′ 45.5″	21.0	11° 20′ 46.3″
44.0	21.7	46.2
44.8	20.7	46.4
43.7	21.2	45.6
43.9	21.0	45.7
	Mean:	11° 29′ 46.0″
	Error:	±0.3″

Table 3. Ag $K\alpha_1$.

θ_t	t °C	θ_0
13° 42′ 39.7″	22.4	13° 42′ 42.4″
40.3	21.6	43.0
39.6	22.2	42.7
38.6	22.6	41.9
39.5	21.5	42.1
	Mean:	13° 42′ 42.4″
	Error:	±0.4″

double crystal spectrometer determination by Bearden [1], 558.255 XU, one of the five values entering in the mean used by Ingelstam. Sandström has chosen the mean value of the measurements of Bearden and N. Nilsson, which is the value used above. The authors feel, however, that more importance should be attached to Ingelstam's value. His wavelength values of W $K\alpha_1$ and Sb $K\alpha_1$, based on a set of reference lines, compatible with the lower value of Ag $K\alpha_1$, have shown excellent agreement with the

Table 4. Mo $K\alpha_1$.

θ_t	t °C	θ_0
17° 29′ 21.7	24.0	18° 29′ 27.1″
22.2	23.5	27.1
21.7	24.5	27.5
23.2	23.0	27.7
	Mean:	17° 29′ 27.4″
	Error:	±0.4″

Table 5. The spectrometer constant d.

Line	Wavelength, XU	d, XU, with errors
W $K\alpha_1$	208.573	1177.540 ± 0.013
Sb $K\alpha_1$	469.373	1177.544 ± 0.008
Ag $K\alpha_1$	558.253	1177.560 ± 0.009
Mo $K\alpha_1$	707.831	1177.539 ± 0.007

values found by Du Mond [15] and N. Nilsson [11], and close compatibility with the present measurements. If we adopt a wavelength value which is the mean of Ingelstam's and N. Nilsson's values, we obtain:

Line	Proposed wavelength, XU	d, XU
Ag $K\alpha_1$	558.246	1177.546 ± 0.009

This shows good agreement with the mean value of d obtained with tungsten, antimony, and molybdenum. The mean value adopted for the instrumental constant, to be used in future measurements, is the following:

$$1177.542 \pm 0.005 \text{ XU}.$$

5.2. *Measurement of U $K\alpha_1$*

Table 6.

θ_t	t °C	θ_0
3° 3′ 32.1″	21.7	3° 3′ 32.5″
33.2	22.6	33.9
33.4	20.4	33.8
33.7	21.4	34.2
32.8	22.7	33.5
32.6	22.8	33.4
	Mean:	3° 3′ 33.4″
	Error:	±0.3″

Wavelength value: 125.689 ± 0.004 XU
Ingelstam [8]: 125.686 ± 0.010 XU

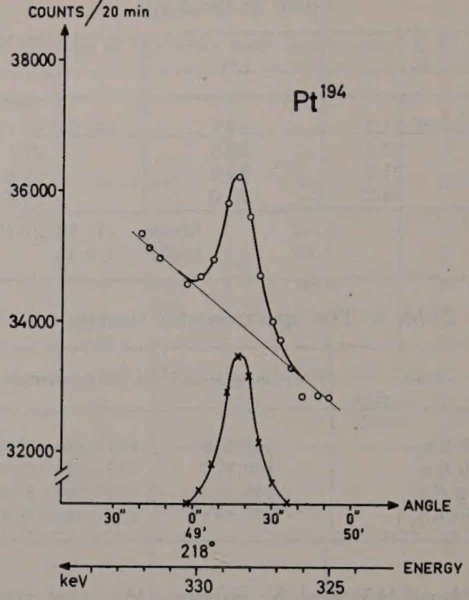

Fig. 9. Gamma transition in Pt^{194}. Line width 15″. Source width 10″.

5.3. *Measurement of a gamma line near the upper energy limit of the spectrometer*

This measurement was performed for the benefit of the nuclear spectroscopy group at the Institute. Energy measurements with a double-focussing beta spectrometer [7] on the conversion lines of a gamma transition in Pt^{194} following the decay of Au^{194} yielded a gamma energy of 328.45 keV. This showed a discrepancy from the value obtained by Ryde and Andersson [13] with the Stockholm curved crystal spectrometer; 329.07 keV, here measured in the decay of Ir^{194}. It was decided to make an attempt with the present spectrometer to measure this gamma line.

An iridium foil, $0.1 \times 3 \times 15$ mm^3, clamped between aluminium plates, was irradiated in the R1 reactor in Stockholm for 64 hours at a pile factor of 5. A total activity of 250 mC of Ir^{194} was obtained. The abundance of the transition is given as 27 %. The 19-hour activity was followed for 24 hours. Two pairs of lines were measured.

Table 7. Pt^{194}.

θ_t	t °C	θ_0
54′ 58.3″ 54′ 59.0″	20.3 20.8	54′ 58.4″ 54′ 59.1″
	Mean: Error:	54′ 58.8″ ±0.5″

Wavelength: 37.663 ± 0.006 XU.
Energy: 328.49 ± 0.05 keV (12372.2 keV · XU).

The total counting rate was initially 1700 counts/min and the signal to background ratio 1:10. This ratio decreased with time because of the presence of the long-lived activity of Ir^{192}. One of the lines is seen in Fig. 9. The line width corresponds to a resolution of 0.45 %.

The result is given in Table 7 and shows good agreement with the beta spectrometer value.

SUMMARY

A curved crystal spectrometer of the Du Mond type for precision measurements of X- and gamma-ray energies from 20 keV to 350 keV is described. The spectrometer utilizes a fixed source and a moving detector, the crystal being rotated and the Bragg angle measured with a precision angular scale. The advantages of this arrangement are discussed. An experimental comparison between two methods of bending the crystal lamina is made.

The instrument is calibrated with the $K\alpha_1$ lines of Mo, Ag, Sb, and W. Measurements of $U\,K\alpha_1$ and a gamma transition in Pt^{194} (328.49 ± 0.05 keV) are reported.

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